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M203 PROPELLING CHARGE RESIDUE INVESTIGATION PART I

D. S. DOWNS L. E. HARRIS K. RUSSELL

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND

LARGE CALIBER

WEAPON SYSTEMS LABORATORY

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of a higher melting point concluded to be the only programs, procedures, and	wax (Indramic 170C) in the wear reducing additive was suitable solution to the problem. Details of all test results are included.

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INTRODUCTION

During safety testing of the M2O3El propelling charge in July 1978, a gross condition of residue was observed for charges which were conditioned at 63°C for at least 24 hours. This residue occurred during firing of the latter portion of a 48-round group. The residue consisted of large portions of non-combusted bag, liner, and jacket material which was either loose in the gun chamber or firmly attached to the chamber wall. The residue was of concern because the quantity was large enough to interfere with projectile seating, and one M549El projectile had failed to seat fully. Other performance parameters for the M2O3El charge were satisfactory, but, because of the residue condition, the charge was not considered safe for production and field issue.

A test program was immediately initiated to determine the cause of the problem and to provide corrective measures. Early in the program it was found that the gross residue condition was related primarily to charges conditioned at 53° and fired in a hot gun tube (71°C and above). As a result of later residue tests, it became apparent that the residue problem was also common to the M203 propelling charges produced since December 1977. Subsequent testing concentrated on the M203 charge.

The testing involved intensive investigations to determine the root cause of the residue problem and to provide a solution. This testing took a period of approximately 6 months, during which several design variations were tested and over 1000 charges were fired. In this report we summarize the tests and point out the salient features of the results. Included are several general observations on the design of separately loaded bag charges and on the optimization and dispersion of the wear preventing additive liner.

BACKGROUND

The M203 propelling charge is the zone 8 charge for the 155-mm M198 towed howitzer and was currently being qualified for the M109A2/A3 self-propelled howitzer. The cannons for these howitzers are the M199 and M185, respectively. Figure 1 shows the charge configuration as of October 1976. The M30Al propellant which is

used in the M203 propelling charge has a flame temperature of approximately 3040 K. When incorporated with the M203 charge and fired in the M199 cannon, this propellant has a maximum operating pressure of approximately 324 MPa (47 kpsi) when fired at 21°C. Because of the high temperature and pressure produced, a wear preventing additive liner is included in the charge to reduce the amount of gun barrel erosion. This liner is similar to that used in the 105-mm tank rounds and consists of a mixture of TiO₂ and wax in the ratio of approximately 45/55 percent by weight. Ignition of the charge is achieved with 142 grams of class 1 black power in a basepad and center-core system. Approximately 454 grams of potassium sulfate are affixed to the forward end of the charge in a pancake-shaped bag, to limit the amount of muzzle flash.

Early in 1977 the M203El, a modified version of the M203 (fig. 2), was introduced. For the M203El, the length of the charge was reduced from 787 mm (31 in.) to 749 mm (29.5 in.) to provide compatibility with the M185 cannon of the M109A2/A3 self-propelled howitzers. As a result, the charge diameter increased from 151 mm (5.95 in.) to 156 mm (6.15 in.). A doughnut configuration for the flash reducer package was introduced to eliminate the potential for non-ignition of the M549Al rocket motor. Lastly, the black power used in the base pad and the core was changed to a faster burning type to minimize ignition delay and hangfire potential.

In the July 1978 safety test, 280 rounds, conditioned at both hot and cold temperatures, were fired, and the residue occurred during the last 8 of the group of 48 rounds being fired after conditioning and after subjection to transportation/vibration tests at 63°C . Typical examples of the residue observed are shown in figure 3.

During the period 1 to 3 August 1978 a residue test established that the M203El residue occurred with the combined conditions of a hot charge and a hot gun tube. External tube temperatures were monitored during this test. On 4 August 1978 Indiana Army Ammunition Plant (IAAP) reported that the source of the wax used in the wear reducing liner had changed between the last production of the M203 (December 1977) and the production of the M203El charges (June 1978). The earlier wax had been purchased in mid-1977 from Industrial Raw Materials Corporation, and representative samples had a melting point of 70.7°C (159.2°F). The wax used in liners for the M203El was received in February 1978 and had a drop melting point of 69.5°C (157.1°F). While both wax batches

¹K. Russell, Trip Report, JPG, 1 August 1978.

were purchased from Industrial Raw Materials Corporation, IAAP later determined that the original sources differed. The February 1978 wax came from a Shell refinery in Japan, whereas the other waxes came from the United States.

A second residue test on the M203El was performed on 24 August 1978^2 . Most of the testing was done when the external tube temperature was above 21°C (71°F). Several variations were tested in an attempt to estabish the cause of the residue. The changes included varying the type of black powder, flash reducer configuration, and charge length-to-diameter ratio. In addition M203El charges with liners made from earlier (old) wax were tested. All variations fired, including the M203, gave unacceptable residue. Two charges fired with an oversize diameter of 165 mm (6.5 in.) left an extremely large amount of residue.

ROOT CAUSE ANALYSIS

At this time a more concentrated effort was undertaken to determine the cause of the residue. Two teams were formed to perform a detailed root cause analysis and to establish test plans based on this analysis. For the root cause analysis each team accumulated and reviewed all background information, including both production procedures and test firing data. Following the review a brainstorming session was conducted to list all possible causes, independent of ranking as to probability. (In this procedure some of the causes were, intentionally, highly speculative.) The teams next proposed failure sequences which described how each cause (failure mode) could lead to the formation of residue. The teams prepared a root cause analysis chart for each possible failure mode. A typical completed chart is given in figure 4. charts data in support of and refuting the failure sequence were listed separately and the additional data and tests required to evaluate the cause were noted.

Each possible cause was then rated as probable, likely, or unlikely. The rating process was first done separately by the two teams and then it was discussed jointly to establish the final rankings. Plans were then developed to investigate, on the firing

²K. Russell, Trip Report, JPG, 24 August 1978.

range and in the laboratory, the probable and likely causes. From this time, the two teams operated as a single unit.

Table 1 lists the possible root causes of residue which were proposed and their rankings. These causes are grouped under the general headings of configuration, wax properties, firing conditions, and material variations. Some of the root causes refer to configurational variations between the M203 and M203El charges. (At the time of the first root cause analysis it was not clear that the M203 charge would produce residue. This fact was established later.) Table 2 compares the properties and characteristics of the M203 and the M203El propelling charges.

TESTING

Development of Test Logic

A fundamental question was raised by the results of the 24 August 1978 residue tests where residue was observed from the current production of M203 charges: "Is the residue associated only with charges fabricated from current production materials and propellant, or is the residue also associated with charges fabricated during the R&D cycle?" Although thousands of charges with the M203 configuration had been fired during the R&D cycle, residue had not been a problem then. A test logic outline (consisting of plan A and plan B) was developed based on possible answers to this ques-Plan A involved firings designed to answer the tion (fig. 5). Plan B included both a propellant production inabove question. vestigation and a materials investigation and was contingent on the Plan B consisted of both residue tests and outcome of Plan A. laboratory work.

In hindsight, the residue problem almost certainly existed before July 1978, although the frequency and severity may have been lower than in July 1978. Some previous testing had occurred with the hot charge/hot gun combination. However, much of the high-rate-of-fire (hot gun) testing was with ambient temperature charges or was under test conditions that made it difficult to observe the chamber closely. Hot-charge tests were frequently done in cold months or with a number of rounds less than that required to heat the cannon to the temperature threshold where gross residue is produced. These aspects, together with the fact that test observers were not oriented at that time to critically look for

residue, are thought to be the reasons the problem did not become evident sooner in the M203 development cycle.

In various reports of firing tests from 1973 to 1977 there are occasional notes and photographs of a similar residue. However, the frequency was low, and there were no reported instances of failure to fully seat a projectile because of residue.

Results of Plan A

The results of plan A [which used both R&D and production rounds and which was conducted on 6 October 1978^3 at Jefferson Proving Ground (JPG)] are shown in table 3. A series of M203 charges with the wear preventing additive liner removed were fired to heat the tube to 77° C. A rate-of-fire was maintained which held the gun temperature in the range of 71-to- 82° C. Three cleaner rounds (also M203 charges without liners) were fired between each test series. All test charges were conditioned at 63° C. After each firing in a test series, the chamber was examined for residue, and the size and location were recorded if it occurred. All residue was noted, independent of size and amount. The charges without liners did not produce residue, but new production charges (lot 78J-M203) produced residue in every round fired.

One charge lot (IS-033-77L, fabricated from current production materials but with R&D propellant) was made by downloading charges from lot 77L-M203 and reloading them with propellant from lot E36. One small piece of residue was observed in the 15 rounds fired. This result raised the question of possible changes in the production of the M30Al propellant.

Table 4 compares the characteristics of the propellant lots used in the M203 charge over a 4-year period. One significant process variation is the conversion to a continuous nitration process. (Another significant variation was discovered later — the effect of downloading and reloading on the mechanical integrity of the wear preventive additive liner and, ultimately, on the production of residue. This variation is discussed later in the report.)

Results of Plan B

After analysis of the results of plan A, plan B (to investigate propellant variations in production and materials variations) was begun.

³K. Russell, Trip Report, JPG, 6 October 1978.

Two general pass/fail criteria were proposed for reaching a solution to the problem: When the M203 or M203El propelling charge is fired under any actual or simulated condition of service use:

- 1. There should be no cloth residue adhering to the chamber wall.
- 2. There should be no failure to fully seat a projectile due to residues from the propelling charge.

These criteria were to be assessed with the view that some propelling charge residue was expected at a low frequency and in small sizes and that no propelling charge leaves the chamber completely clean. There was no ready way to quantify the results of the tests; thus, the resolution of the M203/M203El residue problem will necessarily be partially subjective.

Initial Materials Investigation

Procedure

Because the results of plan A had shown that charges without wear preventive additive liners (cleaner rounds) did not produce residue, considerable attention was focused on the properties and configuration of the liner.

A closed bomb test was conducted as follows. The closed bomb was heated to 71°C. Pieces of liner were placed in the bomb with 155 mm M30Al propellant to determine whether the liner would be consumed. The amounts of liner and propellant were chosen to be in the same weight proportions as in the M203 charge. In some tests the liner fragment was left loose in the bomb, and in others it was intentionally stuck to the wall. Residue consisting of the liner essentially intact was recovered for both conditions. Wax impregnated jacket material was also tested, both loose and stuck to the wall, and was consumed in each case.

An erosion test fixture (which is basically a closed bomb fitted with a barrel) was also used for similar tests of liner fragments and jacket material. However, the fixture was not preheated. When the liner fragment was loose, some residue was ejected; when the fragment was stuck to the wall, some residue remained in the bomb. As in the closed bomb test above, wax impregnated jacket material was consumed when either loose or stuck to the walls.

An idealized heat flow analysis was performed to simulate the conditions that the liner might encounter during an actual gun firing. The simulation model involved the immersion of a 1.2-mm slab of the ${\rm Ti0}_2/{\rm wax}$ liner, which was initially at a temperature of 300 K, into a 3000 K temperature bath for 12.2 ms. Convective heating was not included. The calculation indicated that under the above conditions the center of the slab barely reached its 344 K melting point after 12.2 ms.

The results of the closed bomb and erosion fixture tests and the heat flow analysis strongly suggested that physical breakup and convective heating are required to consume and disperse the liner. In actual gun conditions, presumably the necessary conditions are provided by the forward gas velocity and turbulence.

A residue test based on materials variations was developed with several key factors in mind. Previous tests had indicated the possibility that a buildup or foundation of some sticky deposit on the chamber wall was necessary before the cloth began to adhere and residue was observed. This sticky deposit was thought to arise either from the swabbing procedure and/or from liner wax buildup on the chamber wall.

Another hypothesis for a source of sticking residue was that the wax, or some fraction of the wax, wets the bag and jacket material during the 63°C conditioning and provides the glue which causes the cloth to adhere to the chamber wall when forced there during the firing.

Variations of the liner materials and configuration were based on the failure of the liner to be consumed in the closed bomb tests and on previous experience with liner positioning in 105 mm tank ammunition where it was found that moving the liner forward eliminated residue.

On 1 and 2 November 1978 the first of two residue tests of materials variations was conducted. M203 charges from 1ot 78J-69806, conditioned at 21°C, were used to heat the tube to 71°C. On day 1, the chamber was swabbed after each round, and on day 2 the chamber was not swabbed. The purpose was to determine the effect of swabbing and to determine whether the swabbing procedure had been a factor in residue observed with the M203E1 charge during the product verification test (PVT) at APG. PVT for the M198 howitzer was ongoing at APG in late 1978. Approximately 4000 rounds had been fired using both M203 and M203E1 propelling charges. The practice of swabbing was generally followed and residue was observed intermittently.)

Cleaners (M203 charges without liners) were fired after each test group to provide similar initial conditions for each series. The baseline, residue-producing charge was the M203, lot 78J-69806. Instrumentation included a thermocouple for external tube temperature measurement, copper crusher gages, piezo-gages for pressure-time measurement at two locations to obtain differential pressures, coils for velocity measurement, and heat sensors. span Corporation installed the heat sensors and provided data reduction to determine the heat input per unit area at a position These measurements were used as a near the origin of rifling. basis for comparing the potential of the various modifications for producing tube wear relative to the baseline charge. All charges were conditioned at 63°C before firing.

After each round was fired, the chamber was inspected and all residue noted. If sticking residue occurred, the chamber was cleaned and scraped before the next round.

Detailed descriptions of the charges fabricated for the materials variations tests are given in appendix A.

Results.

Table 5 summarizes the results of the tests performed on 1 and 2 November 1978 at YPG. (Testing had been moved from JPG to YPG, to insure, as much as possible, conditions that would favor maintaining the high tube temperature required by the test plan.) Promising variations for eliminating or reducing residue were:

- 1. Wear Liners with Indramic 170C.
- 2. Standard liners in mylar envelopes or baggies, relocated between the bag and the jacket.
- 3. Half-length liners positioned at the forward end of the charge, either single or double thickness.
 - 4. No wear liner.
 - 5. Lighter weight jacket fabric.
- 6. Old charge materials with new (lot 69806) propellant.
 - 7. Charges with old (lot El4) propellant.

It was observed that 21°C conditioned charges did not produce residue either with or without swabbing, although the

chamber did appear cleaner with swabbing. From this result it was concluded that swabbing was not a factor in producing residue.

Variations which had no apparent effect for reducing residue were:

- 1. Elimination of the dacron staple.
- 2. Increased charge weight (to produce higher chamber pressure).
- 3. Red-dye in jacket fabric with doughnut-configuration flash reducer package.
 - 4. Elimination of the gusset, tie straps, and boot.
 - 5. Jacket fabric with polyvinyl alcohol finish.
- 6. Lot 78J-69806 of M203 propellant conditioned at 63°C .

Two additional materials variations were tested during the residue test on the propellant pilot lots (discussed below). One variation was charges with the standard liner inclosed in mylar and with liner relocated between the jacket and bag. These charges were fabricated by adding the liner to cleaner rounds on hand at YPG. Residue appeared in 10 of 10 rounds fired (table 6) as compared to the 4-in-11 frequency observed on the 1 November 1978 occasion. (It was later realized that this series contained twice the normal amount of lead foil, effectively in a double layer, because the lead foil was previously included in the cleaner rounds. This variation was, therefore, retested on a later occasion with a single lead foil.)

The other variation was a standard M203 charge down-loaded and reloaded, with the liner intentionally weakened during the process 4. Our purpose was to test the hypothesis that the reloading process damages or physically weakens the liner, providing for easier breakup during the firing cycle and for consequent reduction in residue. No residue was obtained with the five charges fired with this variation. A summary of the materials/propellant variations is shown in table 7. Physical damage to the liner appears to be a factor and must be considered in the interpretation

⁴A group of five charges were prepared and fired during an on-going test on 1 December 1978.

of the residue results. There is some question as to the amount of handling experienced by the lot PAE-09692 (table 3), since it was manufactured by reuse of main charge components from an older version of the charge. The remanufacture involved replacement of igniter components. Thus, while not reloaded as part of these tests, the charge lot did see extra handling in the past. Based on these results it is very possible that charges fabricated from old materials/old propellant would produce residue in the hot charge and hot tube conditions of these tests.

Propellant Production Investigation

On 18 October 1978 a visit was made to Radford Army Ammunition Plant (RAAP) for the purpose of ascertaining details of the propellant production for the five propellant lots listed in table 4. These lots had been used in the M203 propelling charge. Several variables were identified for the 1977 lots:

- 1. CIN (continuous) vs batch process for cellulose nitration.
- 2. Higher percent nitrogen in batch process nitrocellulose.
 - 3. Toluene vs benzene as the denaturant in the alcohol.
 - 4. Source change for ethyl centralite.
- 5. The percentage of NC to NG is lower in the newer lots, with a corresponding increase in the percentage of nitroguanadine (NQ) $^{\circ}$

Also, the relative quickness (RQ) (based on lot E14 as standard) was somewhat lower for these three newer production lots, even though the lots were granulated in the same die sets as the earlier production lots. The temperature coefficients for these newer lots were also lower (fig. 6). In addition, the higher porosity observed in the earlier lots is not as evident in the newer lots.

The observation on porosity is based on in-plant quality control records at RAAP and on a procedure that RAAP personnel use to determine a quality score based on inspection of grain sections under magnification. From production experience they know that voids can be induced by extruding solvent rich at lower pressure and faster strand rates or by adding water to the mix (usually as increased moisture in nitrocellulose after dehydration). The three

lots produced in 1977 have a better quality core -- and therefore fewer observed voids than the propellant made in 1973 and 1974.

A principal cause of the reduction in RQ for the newer lots was attributed to the reduced porosity. Since the newer production process appeared to be producing a more consolidated, low porosity grain (particularly after the introduction of the CIN process), a pilot lot series of five process variations were proposed in an attempt to re-introduce the higher porosity and to reproduce propellant like that used in the R&D cycle.

Figure 7 shows five pilot lots of propellant proposed as possible improvements to the 1977 production. In each column the propellant was extruded through the same die sets. Reading down the column, RQ increases because of the composition or process differences. Reading across the rows, RQ increases due to decreasing web caused by die differences. All five pilot lots used nitrocellulose with a percentage of nitrogen above 12.60 and with increased NG/NG and decreased NQ. The intent was not to make the pilot lots more like the 1974-and-prior propellant lots but to stay within the specification limits for composition.

The five pilot lots were intended to give higher porosity products. For lots A-1, A-2, and A-3 the solvent level and process controls were the same as in the regular production. However, the B-1 and B-2 samples were over-solvated and were extruded at lower pressures.

 $$\operatorname{\textsc{The}}$$ measured web and RQ of the five pilot lots shown in table 8 are consistent with planned results.

M203 charges loaded with the pilot lots were fired during tests at Yuma Proving Ground (YPG) on 28 to 30 November 1978. Table 8 also shows the charge weight assessed to produce the required velocity and the resultant peak chamber pressure. (Lower charge weights and higher pressures are consistent with the increased RQs and with decresed web sizes resulting from die changes and processing.)

To evaluate the effect on residue, each pilot lot was fired at the assessed weight. Lot 78J-69806 was used as a baseline, residue producing charge. Under the conditions of $63^{\circ}C$ conditioned charges fired in a hot gun tube, each pilot lot produced some residue (table 6).

Lot A-l produced significantly more residue than the other four lots. The indication was that the residue problem might be alleviated by changing the process variables to achieve an RQ

comparable to the earlier, R&D propellant. However, the problem would certainly not be resolved because all pilot lots gave some amount of residue with every round fired.

Figure 8 shows the pressure-temperature data for the pilot lots and for the baseline lot. The temperature coefficients for the propellant pilot lot firings are more comparable to the earlier R&D propellants than to product on propellants. The cause of the variation in temperature coefficient is not well understood.

In addition to the process variations described above, independent laboratory studies of the propellants were also undertaken (app B). These include Electron Spectroscopy for Chemical Analysis (ESCA), Scanning Electron Microscopy (SEM), x-ray and neutron diffraction, and mechanical properties tests.

Several characteristics of propellant lots E36 and 69805 were compared. The density and heat of explosion were found to be essentially the same. No differences in propellant composition were found using the sensitive ESCA technique. Compressive strengths and corresponding strains were found to be essentially the same. Neutron diffraction data revealed a noticeable, relative change which suggested the possibility of differences in the amorphous component of the batch produced NC and the CIN-processes NC. Scanning electron micrographs of broken grains showed variations in the morphology of the NQ; however, these variations appeared in both lots.

Follow-On Materials Investigation

Procedure

After analysis of the results of the preceding laboratory and firing tests, the general conclusions were that the residue problem could be resolved by:

- l. Using a high melting point wax (Indramic 170C) in the wear reducing additive liner.
- 2. Physical weakening of the standard wear reducing additive liner.
- 3. Shortening the liner to half-length and positioning it forward in the charge.

The tests had also shown that the residue problem was alleviated somewhat by inclosing the liner in mylar and relocating it between the bag and jacket. Also, charges produced with the pilot

propellants had yielded smaller amounts of residue, although the frequency was not significantly decreased.

At this time the emphasis was strongly focused on modifications to the wear liner. A second materials variation test plan was developed with the primary variables being the melting point of the wax, liner length, and gridding. [Gridding represents an effort to systematically weaken the liner by scoring with 25.4 mm (l in.) squares. Gridding was accomplished by drawing a circular blade (pizza-cutter) across the liner without cutting through the scrim or the lead foil.] Secondary variables were inclosures of the liner in mylar and its relocation between the bag and jacket, and varying of the liner thickness.

Instrumentation was the same as in the previous materials test, and some of the most promising charge variations were tested with both 294 K or 336 K preconditioning. In the November 1-2 tests the heat sensor was at 1.02~m (40.25~in.) from rear face of the tube (RFT). In the December 14-16 test sensors were located at both 1.02~m (40.25~in.) and 1.06~m (41.7~in.) from RFT.

Results

Table 9 summarizes the residue test results obtained on 14-16 December 1978. Detailed descriptions of the materials variations are given in appendix A. Of the 63°C charges, only the series with Indramic 170C wax was completely free of residue. (The half-length liner variations, fired on 1 and 2 November 1978, were residue free but they had shown considerably higher average heat inputs than the baseline charge and were therefore not retested.) Three-quarter and seven-eighth length liners and three-quarter thickness liners all produced residue and had average heat inputs higher than the baseline groups.

Comparisons of the average heat inputs must be made with caution (ref 1). Because of the large number of rounds fired and the use of charges without liners, the gun tubes wore considerably. During the November 1-2 and December 14-16 tests the tubes used were worn from approximately 60% life remaining to near condemnation. As a result, the average chamber pressure for similar charges firing similar projectiles became lower as the test proceeded, and the average heat inputs decreased as well. This fact is illustrated in table 10. Since the materials variations residue tests were instrumented for heat input, the opportunity was taken to assess the role of the projectile-type on the average heat inputs. A decreasing trend in both average heat input and chamber pressures was noted over the 3 days of testing in December.

Table 11 presents the heat input data for the baselines and Indramic 170C wax liners along with the average chamber pressures. All of these tests used inert M101 projectiles. Since the average heat inputs should be compared (along with the average chamber pressures), the results for the Indramic 170C wax were very encouraging. The 1-2 November and 14-16 December tests indicate that the performance of the Indramic 170C wax was comparable to the wax in the baseline charge at both 21°C and 63°C. In fact, since the slightly higher values of the heat inputs associate with considerably higher chamber pressures, the Indramic 170C wax may be slightly more effective.

PRELIMINARY AND FINAL ACCEPTANCE TESTS

Based on the above results the conclusion reached was that the use of Indramic 170C with a higher melting point (82° C instead of 71° C) wax in the wear reducing additive liner eliminated residue under the hot charge/hot gun tube conditions tested. It was recommended that the M2O3 charge be released for production. In addition to Indramic 170C wax in the liner, three other modifications were included in the technical data package for the production lot:

- l. Incorporating a doughnut flash reducer and off-center positioning of the tie strap knots to insure reliable ignition of the RAP round, and to maintain charge length limits.
- 2. Dyeing the lacing jacket red and marking it as 8S to facilitate identification and to distinguish the M203 from the M119Al charge.
- 3. Establishing a maximum length of 768 mm (30.25 in.) to permit use in the M185 cannon (M109A2/A3 self-propelled howitzer).

These additional modifications had been tested to some extent in the residue tests, and in earlier PVT and safety tests. Table 12 shows that there was no residue during preliminary acceptance testing of an early sample from the M203Al charge, lot 79A-69807, which has the three above features.

The final acceptance test on lot 79A-69807 was fired on 9 March 1979 and is summarized in table 13. Sixty rounds were fired at various temperatures. Two small pieces of cloth residue were observed, one at $-51^{\circ}C$ and one at $63^{\circ}C$, both less than 13 cm^2 .

These small pieces of residue were considered to present no problem.

Although the Calspan heat input measurement has not been established as an accepted criterion for predicting wear life of the cannon, a correlation apears to exist between heat input and wear, particularly for design variations of a charge firing similar projectiles under similar conditions. The materials variations residue tests indicate that the heat input from charges using high melting point wax (Indramic 170C) is certainly no higher than from the baseline M203 charge.

On the occasions where the Indramic 170C wax was tested in charges preconditioned to 63°C (table 9), residue was produced by test groups fired both immediately before and immediately after the charges with the indramic 170C. No residue was observed with the Indramic 170C. The total number of rounds fired at this point represented a relatively small sampling. This result was considered to be significant since the firing conditions were such that the preceding and succeeding variations gave residue.

The Indramic 170C wax rounds, conditioned at 21°F, tested on 14 December, gave residue in the tube in two of fifteen rounds. On one of these occasions a small (25 mm by 76 mm) piece of cloth residue was found in the chamber and removed. The residue in the tube was shot out with the succeeding round. This type of loose residue was judged to present no problem to projectile ram and chambering.

BALLISTIC PERFORMANCE

For essentially every round fired in the various firing tests discussed thus far, there was full instrumentation for assessing interior ballistic performance. These data have not been addressed in detail in this report since the focus is on the residue problem. However, the data were continually used to determine what, if any, effect the charge variations tested might have on ballistic performance. Also the data were used as a basis for final acceptance of the M203Al charge design, which evolved from this work. The data base is significant - approximately 1400 rounds. Generally, good performance of the M203El and M203 propelling charge was observed, regardless of the variables of charge construction. The only exception was high differential pressure indications on a few charges which were intentionally weakened by breaking up the wax/TiO₂ liner or downloading and reloading.

CONCLUSIONS

A residue problem was defined for a relatively narrow range of conditions: namely, the M203/M203El charges conditioned at 63°C and fired from gun tubes whose external temperature at the position of the origin of rifling was between 60°C and 93°C. A root-cause analysis procedure was adapted to identify the cause and to determine a suitable fix. Causes were identified and subsequently plans for both laboratory investigations and gun firings of modified M203 charge were generated. A procedure was established, in the form of a logic diagram, to serve as a guide to the overall gun firing test program. Because of uncertainties in the early test results, variations in both charge component materials and propellant production procedures were pursued as possible solutions to the residue problem.

As a result of the residue test program, the following general observations can be made:

- l. Instrumenting the tests as fully as possible proved to be very important. Pressure, velocity, temperature, and heat input data were all used in the assessment of the various charge modifications tested.
- 2. A substantial data base was established on which it was possible to assess the effects of chamber pressure and projectile type on the interpretation of heat input data. In addition, it was established that the present liner configuration (length/thickness) is essentially optimized. These results have been published in a separate report (ref 1).
- 3. It is clear that we do not have a good understanding of how the inert components of the propelling charge are consumed (or not consumed) and of how the wear preventing additive liner is actually dispersed. From the laboratory data and theoretical studies, turbulence and convective heating produced by the forward gas velocity appear to be essential. Also, from firing data, the worst instance of residue was seen with an M203El charge with oversize diameter. This result indicates that ignition dynamics and annular ullage (or proximity of the charge to the chamber wall) may be important.
- 4. The propellant processing studies surfaced the problem of the propellant temperature coefficient. This coefficient is now

the subject of a separate study. Propellant variations did not affect the frequency of residue.

5. Lastly, the melting point of the wax was indicated as an important parameter in the formation of residue when the charges were conditioned at 63° C and fired in hot gun tubes.

RECOMMENDATIONS

Substitution of a higher melting point wax (Indramic 170C) in the wear reducing additive liner was recommended as the only acceptable solution to the problem. Charges produced with this liner modification gave no residue under the conditions tested when con-Heat inputs to the gun tube with this liner ditioned at 21°C. modification were judged to be equivalent to or slightly lower than those with the liner in the baseline M203 charge. Therefore, it was concluded that the wear and erosion characteristics would not be affected. Based on the recommendation, the technical data package was revised, and the M203Al charge was produced with a high melting point wax, Indramic 170C, in the wear preventing additive Three other minor modifications were included in the revised technical data package. These changes included modifying the flash reducer package configuration from pancake to doughnut and repositioning the tie knots, specifying a 768 mm maximum length, and using the red-dyed jacket with 8S designation.

A residue problem appeared again in April 1979 during weapon tests to establish firing tables for the M109A2 howitzer using the mcdified M203 charge. Residue occurred with ambient charges fired in hot gun tubes.

A description of the new problem and the subsequent investigation is the subject of a separate report (ref 2).

REFERENCES

- 1. D. S. Downs, J. A. Lannon, S. Axelrod, C. Gardner, G. Sterbutzel, D. Adams, and F. Vassallo, "Wear Additive Optimization Studies for the High Zone Charge in the 155 mm Artillery System", Proceedings of the 1979 JANNAF Propulsion Meeting, vol 1, CPIA Publication 300, March 1979, p 283.
- D. S. Downs, D. Ellington, L. E. Harris, and K. Russell, "M203 Propelling Charge Residue Investigation, Part II", ARRADCOM Technical Report ARLCD-TR-80007, ARRADCOM, Dover, NJ (in press).

Table 1. Possible causes of residue

Causes	Probability
Related to configuration	
Wear preventing additive liner too close to base of charge.	Probable
Black powder burning rate too fast to melt wax.	Probable
Smaller annular space inhibits combustion on outside of jacket.	Probable
Faster black powder causes more uniform ignition, which forces jacket to wall.	Probable
Center hole in flash reducer vent gases out of forward end, reducing combustion on outside of charge.	Unlikely
Related to wax properties	
Wax softens at 336 K, leading to round-to-round buildup of wax, which causes cloth to adhere to gun chamber.	Probable
Lower melting point wax leads to wax flow into cloth	Probable
Wax exudate impedes cloth burning.	Unlikely
Related to firing conditions	
Rate of fire causes increase in tube temperature, facilitating adhesion of residual wax.	Probable
Swabbing introduces water into gun chamber, which inhibits cloth combustion.	Likely
Swabbing introduces material which causes cloth to stick to wall.	Unlikely
Uncontrolled temperature in conditioning leads to wax melting and migrating into cloth.	Unlikely

Table 1. (cont)

Causes	Probability
Related to material variations	
Cloth too strong and dense, impeding gas penetration and combustion.	Probable
Scrim in wax has variable position, leading to undesirable increase of liner mechanical integrity.	Likely
Thickness of liner inhibits melting of wax.	Unlikely
Adhesive on lead foil applied too thickly.	Unlikely
Water repellant on cloth allows wax to wet cloth, impeding combustion.	Unlikely
Fast burning lacing cord allows jacket to expand to wall, impeding combustion.	Unlikely
Thickness of lead in liner inhibits wax softening.	Unlikely
Propellant not producing high enough pressure.	Likely

Table 2. Properties and characteristics of M203 and M203E1 propelling charges

Properties and characteristics	M203El charge	M203 charge
Propellant	26 1b M30A1	26.1 1b M30A1
	(.080MP)	
	2.03 mm Web,	(2.00 mm web,
	multiperf)	multiperf)
Prop RQ	96.5%	100.0%
Base igniter	1.0 oz Cl l blk pdr	Same
Central igniter	4.0 oz Cl l blk pdr	Same
Blk pdr lot	GOE	CIL
Tube	NC tube w/paper cap	Same
Cloth f/incr. bag and base igniter	Resin-impreg visc rayon, Cl-2	Same
Lacing jacket cloth	Acrylic-visc rayon, C1-3	Same
Central igniter cloth	Resin-impreg visc rayon, C1-3	Same
Wear additive	17.5 oz TiO ₂ /wax matted on dacron serim (0.050 oz/sq in.)	Same
Lead	5.5 oz sheet adhered to serim face (on edge)	Same
Flash reducer		
Material	16 oz K ₂ SO ₄	16 oz K ₂ SO ₄
Construction	Doughnut-type, located	Circular pad
	under tie straps	located under
	•	tie straps
Cloth	Polyester-visc, rayon, Cl-6	Same
Charge dia (max)	6-3/8 in.	6-1/8 in.
Charge length (max)	749 mm (29.5 in.)	774 mm (30.5 in.)

Table 3. Results of plan A

Charge lot	Propellant lot	Rounds fired	Remarks
IS-032	69806	53	To heat tube (no liner)
PAE-09692	E14	14	Fabricated during R&D cycle
77L-M203	69805	15	Current prod, matl's, and prop
IS-033-77L	E36	15	Current prod, matl's, w/R&D prop
78J-M203	69806	10	Current prod
78F-M203E1	69805	8	Tube temp. lower (69-71°C)

Characteristics of M30Al propellant lots used in the M203 propelling charge Table 4.

Pressure MPa (Kpsi)		325 (47.1)		327 (47.4)
Chg wt (kg)		11.90		11.87 11.85 11.80
TV (%)	cess	0.23 0.35	tion process	0.33 0.34 0.26
RQ at 32°C (%)	Benzene denaturant and batch process	100	sene denaturant and continuous nitration process	96.5 96.4 96.2
WEB (cm)	Benzene denat	0.202 0.199	e denaturant a	0.203 0.202 0.202
Date		1/73	Toluen	7/27/77 8/11/77 8/19/77
Lot no.		E14 E36		69805 69806 69807

Results of 1 and 2 November 1978 materials variation residue test Table 5.

Kemarks		Swabbed between RDS	Baseline	High MP wax (Ind 170 C)	No dacron staple	Mylar, liner relocated	Short liner/single thick	Short liner/single thick	Baseline	Cleaners (no liner)		No swabbing	Baseline	Old mat1's/69806 prop	Increased charge wt (M483	projectile)	Ked jacket, doughnut flash reducer	Current prod matl's	doughnut flash reducer/E-14 propellant	No gusset or tie straps	Light-weight fabric	PVA-finish fabric	Baseline	Cleaners (no liner)
Clean	0	30	2	15	0	0	14	9	0	15		22	2	14	0		0	က		0	2	0	0	23
Brown	November)	0	5	0	4	7	1	0	0	0	November)	9	4	0	0		0	9		0	7	1	2	0
Residue	Day 1 (1	0	∞	0	9	7	0	0	4	0	Day 2 (2	0	10	7	9		7	9		7	9	5	2	0
Fired		30	15	15	10	11	15	9	4	15		28	16	15	9		7	15		4	15	9	7	23
T (K)		294	336	336	336	336	336	. 336	336	294		294	336	336	336		336	336		336	336	336	336	294
Charge lot		787-69806	787-69806	IS-034	IS-035	IS-036	IS-038	IS-037	787-69806	1S-046		787-69806	787-69806	M-1	IS-045		IS-043	1S-044		IS-040	IS-042	IS-041	787-69806	1S-046

Table 6. Results of pilot lot residue test

Lot	Rounds fired	Residue	Brown spots	Clean
Baseline (78J-69806)	14	5	6	3
A-1	10	10	0	0
A-2	10	6*	4	0
A-3	10	8*	2	0
B-1	10	10*	0	0
B-2	5	4*	1	0
Reload (78J-69806) Relocate/mylar	5	0	1	4
(double lead)	10	10	0	0

^{*}Amount of residue was significantly less than observed in previous tests.

Table 7. Summary of materials and propellant variations

Remarks	Current prod matl's/ old prop (reloaded)	<pre>0ld matl's/current prod prop (reloaded)</pre>	<pre>Current prod matl's/ old prop (not reloaded)</pre>	Old matl's/old prop (not reloaded)	Current prod matl's/ current prod prop downloaded and re- loaded; no compon- ent changes; liner intentionally weakened.
Residue	1/15	1/15	6/15	0/14	5/0
Fired	15	15	15	14	5
Propellant	E-36	90869	E-14	E-14	90869
Date fired	10/6/78	11/2/78	11/2/78	10/6/78	12/1/78
Charge lot	IS-033	M-1	IS-044	PAE-09692	78J-69806

Table 8. Characteristics of M3OAl propellants pilot lots

			Chg wt	Pres	sure
Lot	Web (mm)	RQ (%)	(kg)	MPa	(Kpsi)
A-1	0.198	98.6	11.82	324.5	(47.1)
A-2	0.192	101.1	11.53	335.5	(48.7)
A-3	0.188	102.0	11.52	339.6	(49.3)
B-1	0.193	100.2	11.69	334.8	(48.6)
B-2	0.189	101.7	11.56	338.9	(49.2)

Results of 14 through 16 December 1978 materials variation residue test Table 9.

Kemarks		HI MP wax (Indramic 170C)	Std wax/3/4 length	Baseline (M549)	Hi MP wax	Hi MP wax/grid	Hi MP wax/grid/mylar/relocated	Std wax/grid	Baseline (M483)		Std wax 7/8 length	Std wax/grid/3/4 length/mylar	Baseline (M483)	Std wax/grid/mylar	Std wax/grid/mylar/relocated	Std wax/3/4 thick	Std wax/3/4 length	Baseline (M549)		Std wax/7/8 length/grid/mylar	Baseline (M101)	Baseline (M549)	Hi MP wax	Hi MP wax/grid	Std wax/7/8 length	Std wax/grid/mylar	Std wax/3/4 length	Baseline (M483)	B-2 propellant, lot
Clean		13	13	1	∞	6	0	0	2		15	4	_	0	0	5	1	က			10	-	က	5	2	0	1	0	0
Brown		0	2	4	2	-1	2	2	3		0	5	2	0	0	က	7	3		4	0	2	2	0	0	ന	_	2	0
Residue	December 1978	2	0	5	0	0	3	∞	7	December 1978	0	7	3	9	9	7	0	2	16 December 1978	5	0	5	0	0	7	9	7	7	5
RDS fired	14	15	15	10	10	10	5	10	9	15	15	10	9	9	9	15	∞	80		10	10	∞	5	5	9	6	9	9	5
T (K)		294	294	336	336	336	336	336	336		294	294	336	336	336	336	336	336		294	294	336	336	336	336	336	336	336	336
Lot No.		IS-068	IS-074	IS-067	IS-068	1S-069	IS-072	IS-070	18-067		IS-075	1S-076	IS-067	IS-071	IS-073	IS-078	IS-074	IS-067		IS-077	IS-067	190-SI	IS-068	690-SI	IS-075	9Z0-SI	IS-074	190-SI	IS-065

Table 10. Heat input as a function of projectile (14 to 16 December 1978)

	14 December	1978	15 December 1978	1978	16 December 1978	er 1978
Projectile	$(J/m^2 \times 10^{-4})$ (MPa)	Pressure (MPa)	$(J/m^2 \times 10^{-4})$	Pressure (MPa)	Heat (J/m ² x 10 ⁻⁴)	Pressure (MPa)
M549	119 ± 5	(336 ± 3)	107 ± 8	(319 ± 3)	107 ± 6	(315 ± 3)
M483	120 ± 5	(347 ± 4)	117 ± 5	(345 ± 5)	109 ± 8	(341 ± 2)
M101	1	1	123 ± 6	(354 ± 8)	114 ± 5	(348 ± 5)

Table 11. Comparison of heat input and pressure for charges with high melting point wax* and baseline charges

Туре	<u>T (K)</u>	Heat input $(J/m^2 \times 10^{-4})$	Pressure (MPa)
	1 t	to 2 November 1978	
Baseline Baseline Baseline Baseline Hi MP wax	336 336 336 336 336	113 ± 8 110 ± 6 113 ± 8 110 ± 8 111 ± 8 20 16 December 1978	359 ± 4 350 ± 6 349 ± 3 345 ± 6 360 ± 4
Baseline Baseline Hi MP wax Hi MP wax Baseline Hi MP wax	336 336 336 336 294 294	114 ± 5 123 ± 6 125 ± 6 116 ± 6 121 ± 8 124 ± 7	348 ± 2 354 ± 8 361 ± 3 348 ± 2 310 ± 3 326 ± 4

^{*}High melt wax was Indramic 170C

Table 12. Residue summary for preliminary acceptance tests

Temperature (°C)	Rds fired	Residue	Brown Spots	Rds fired	Residue	Brown Spots
21	14	0	2	15	0	1
- 54	5	$0_{\mathbf{p}}$	0	20	$0_{\mathbf{p}}$	3
63	10	5	1	25	0	1

^aResidue was ejected from muzzle for approximately 50% of all rounds. Three were smoldering for 79A-69807.

 $^{^{}m b}$ For both baseline and test charges at 219 K, one round gave small (50 mm) fabric scraps in chamber or in early part of rifling.

Table 13. Residue summary for final acceptance test

Residue	1 p	0	0	0	1 _e
Rds	15	10	10	15	10
Residue	18	0	1	p6	ı
Rds	15	5	1	10	1
Projectile	M549Al Inert	M549A1	1116FC M483A1	M549A1	Inert M483A1
Temperature (°C)	21	-51		63	

^aSmall pieces of fabric (10 mm) found 1 to 1.5 meters forward into rifling.

 $^{\mathsf{b}}_{\mathsf{A}}$ small 10 x 30 mm piece of body fabric with wax liner loose in chamber (in notch).

^CTube temperature for the total of 35 hot firings ranged from 80°C to 88°C.

 $^{
m d}_{
m The}$ reference charges gave the expected high incidence rate. Several rounds gave very large pieces, for example, 80 x 380 mm and 50 x 360 mm).

eA small (10 x 70 mm) section of the jacket on chamber wall approximately 89 cm forward of rear face

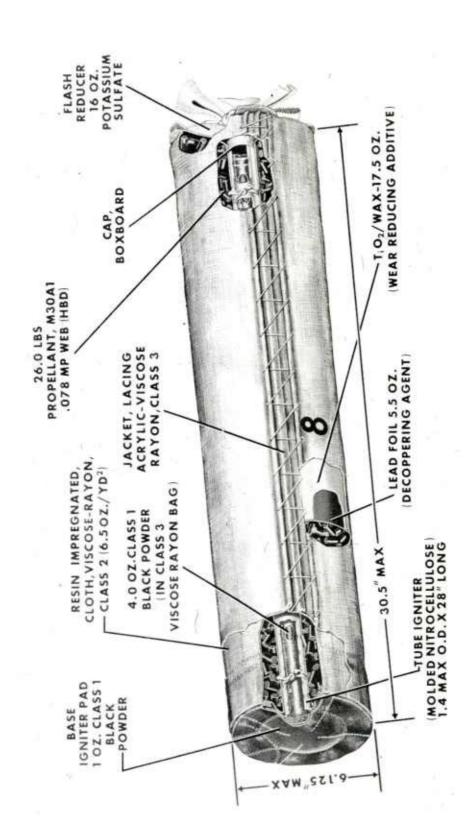


Figure 1. 155-mm propelling charge M203

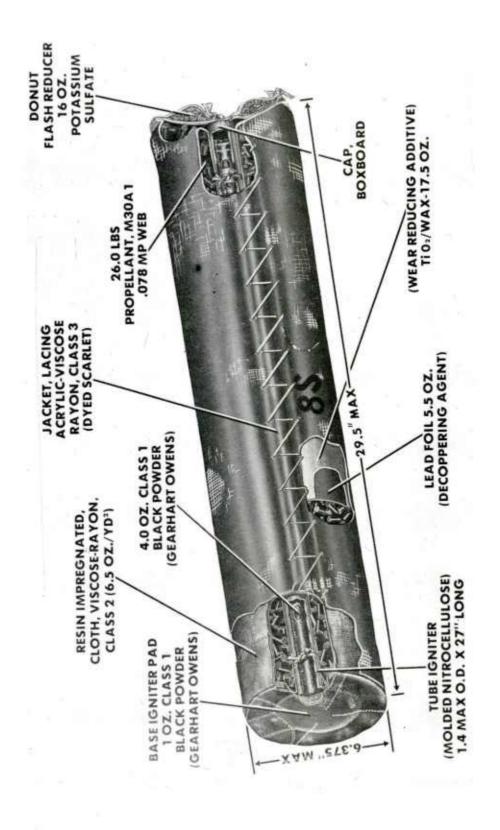


Figure 2. 155-mm propelling charge M203El (red bag zone 8S)



Typical example of residue resulting from safety testing Figure 3.

Dule: 6. Sept 78 Rev. No.: ROO

ROOT CAUSE ANALYSIS CHART

Failure Indication: Residue in Gun Chamber

Cause Probability Estimate:
Probable Cause

and the second commence of the properties of the 1. 145°F preconditioned 2. Characterization of build-up in chamber 'Additional Data wall. Introduced Hot wax in Heated tube by 4. Add MYLAR between liner and bag. 3. Firings with high Tests Required M203E1 without wear additive in 25 round "painting" it on. . M.P. wax. sequence. Did not occur in M203 qualification. EVALUATION Refuting Data 2. Residue usually not observed conditioned at 1450F. Splatter with charges conditioned below 1% wax observed in jacket noted on cartridge case wall. Residue was more likely Residue noted in 105mm 1. Residue was more liker, late in 25 round sequence. 3. Residue noted in 105m M68 tank gun with rounds Supporting Data of conditoned charge. 145°F. τ. fragments normally blown Wax catches unburnt Minimum 20 rounds Wax and some Ti02 conditioned at 145°F. Wax softens at 145°F SPECULATION fired in hot tube. deposited on wall. out during firing. Charges pre-Failure Sequence Failure Mode:

Corrective Action: NONE REQUIRED (Check One)

Conclusion:

Figure 4. Typical root cause analysis chart

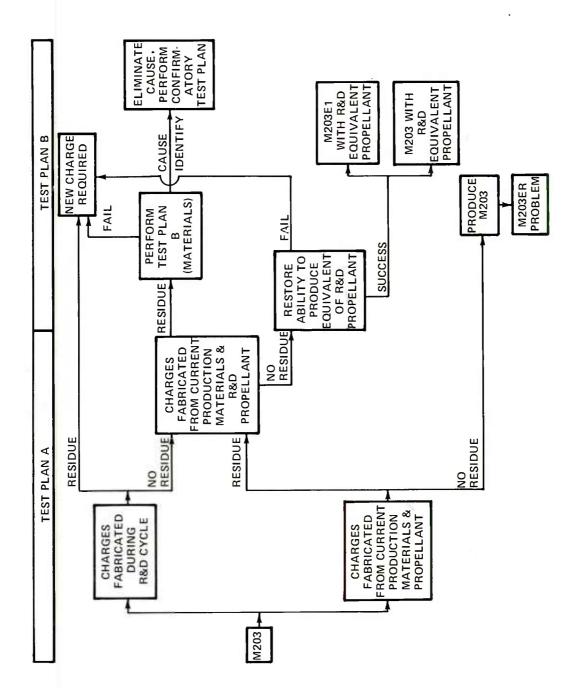


Figure 5. Test logic outline

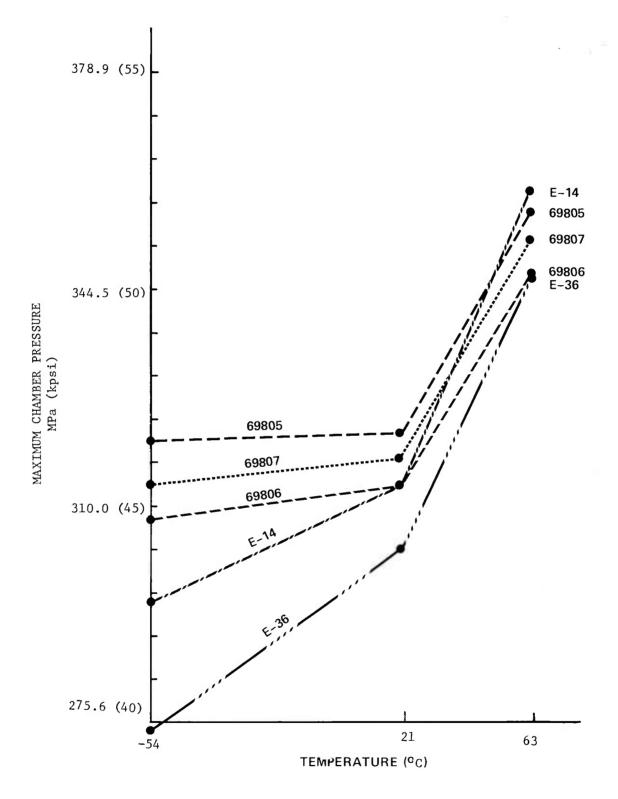


Figure 6. Maximum chamber pressure vs temperature for M203 propellant lots

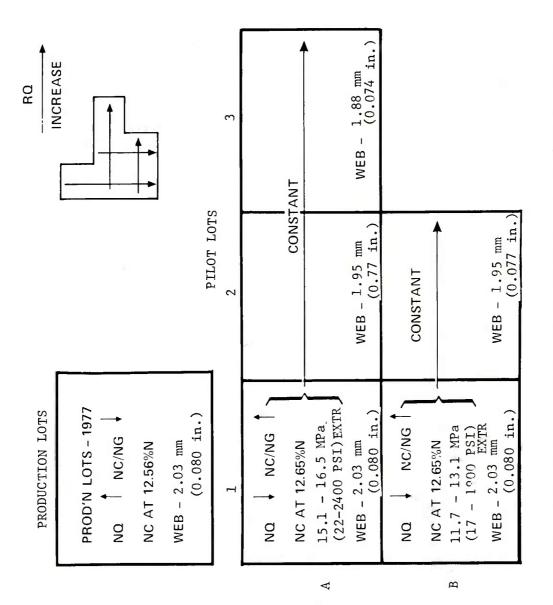
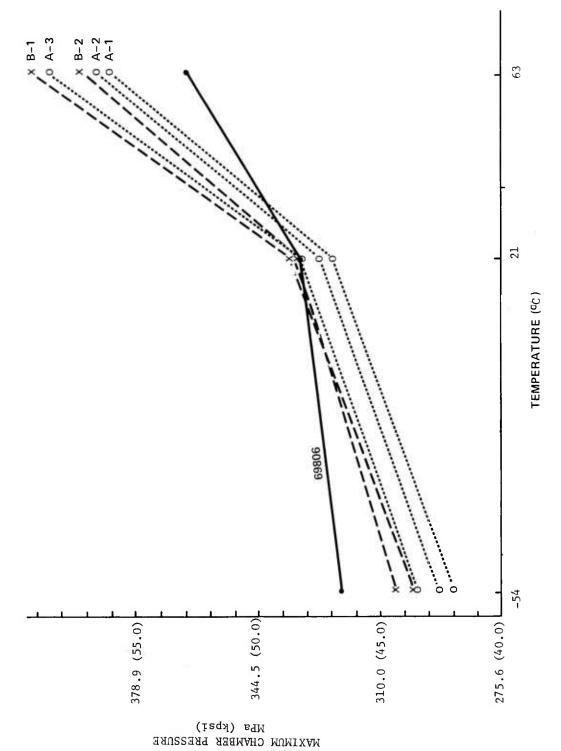


Figure 7. Characteristics of five pilot lots of M30Al propellant



Maximum chamber pressure vs temperature for M203 propellant pilot lots Figure 8.

APPENDIX A

DESCRIPTION OF CHARGES FABRICATED FOR ALL RESIDUE TESTS

Lot number	General description
78F-69805	First production lot of M203El design. Used in Safety Tests at JPG which highlighted the residue problem. (June 1978)
77L-69805	First production lot of M203 design (December 1977)
78F-E36	Made during development trials for M203El. Uses propellant lot E36 from M203 development program.
78H-IS-021	M203 configuration with CIL black powder
78H-IS-022	M203 configuration with Gearhart Owens black powder
78H-IS-023	M203El configuration with Gearhart Owens black powder
78H-IS-024	M203El configuration with CIL black powder
78H-IS-025	M203El using wax purchased in early 1977
78H-IS-026	M203El with a l mil mylar film added to wax/TiO_2 liner on the opposite side from the lead sheeting; i.e., between backing on
78H-IS-027	liner and body fabric M203El with mylar added as in 026, but with the liner assembly reversed so that lead face is next to body fabric and mylar face is inward.
78H-IS-028	A special large diameter, short length charge. OD was approximately 6.5 inches
78J-IS-032	M203 with the entire liner assembly omit- ted. Used as a cleaning charge to purge residual matter from the cannon chamber between test groups.
PAE-09692	XM203E2 (M203) made during development work for M203.
78J-IS-033	M203 Lot 77L-69805 downloaded and reloaded with E36 propellant.

Lot number	General description
78J-69806	M203 lot produced for FOE tests of M198 howitzer systems.
78K-IS-046	Cleaner charges (same as IS-032)
78K-IS-034	First test sample of M203 type charge using the IND 170C, higher melting wax.
78K-IS-035	A standard M203 type charge except that the dacron staple fibers in the wax-TiO $_2$ matrix was omitted.
78K-IS-036	The wax/TiO ₂ liner subassembly was wrapped both sides with mylar, and the entire assembly relocated under the lacing jacket between body assembly and jacket).
78K-IS-037	A standard M203 type charge except that the length of the wax/ ${\rm Ti0}_2$ liner was halved (to approximately 9 1/2 inches)
78K-IS-038	A standard M203 type charge except that the liner was half length (same as IS-037) but the thickness was doubled to maintain the original weight of erosion inhibiting material
78K-IS-039	The same as IS-038 except that E14 propellant was used.
M-1	A test group of the M203 configuration hand made at YPG by down/ordering lot IND-E-172-75 (XM203E2) and reloading with propellant lot 69806
78K-IS-040	M203 type except that the tie straps and boot were eliminated and the gusset between the lacing edges on the jacket was eliminated. The base pad and core igniter assembly was attached to the rear edge of the lacing jacket
78K-IS-041	M203 type with the lacing jacket fabric treated with polyvinyl alcohol

Lot number	General description
78K-IS-042	M203 type except that the lacing jacket was made from a ligher weight fabric
78K-IS-043	M203 type with propellant lot 69806 and the doughnut type flash reducer from the M203El change
78K-IS-044	M203 type loaded the E14 propellant and the doughnut shape flash reducer from the M203E1 charge
78K-IS-045	M203 with propellant charge weight increase of 13 ounces
78M-IS-066	Cleaner charges (same as IS-046 and IS-032)
78M-IS-067	M203 configuration with propellant lot 69807
78M-IS-068	M203 configuration with IND 170C, high melting wax (same as IS-034)
78M-IS-069	M203 configuration with IND 170C high melting wax and the wax surface gridded in linch squares
78M-IS-070	M203 configuration using standard wax, grid-ded in 1 inch squares
78M-IS-071	M203 configuration using standard wax, grid- ded in 1 inch squares
78M-IS-072	M203 configuration using IND 170C high melt wax, gridded in I inch squares, packaged in mylar and the liner relocated between the body and jacket
78M-IS-073	M203 configuration using standard wax, grid- ded in l inch squares, packaged in mylar, and the liner relocated between the body and jacket
78M-IS-074	M203 configuration with the liner assembly trimmed to 3/4 of normal length

Lot number	General description
78M-IS-075	M203 configuration with standard wax, but with the liner assembly trimmed to $7/8$ of usual length
78M-IS-076	M203 configuration with $3/4$ length liner (as in 074) with gridding and packaged in mylar
78M-IS-077	M203 configuration with $7/8$ length liner (as in 075) with gridding and packaged in mylar
78M-IS-078	M203 configuration with standard wax, with liner subassembly at $3/4$ the thickness ($3/4$ weight) of the standard liner
79A-69807	Production lot of M203 charges using IND 170C high melting wax, doughnut type flash reducer, maximum length of 30.25 inch, and with the red dyed lacing jacket marked a zone 8S.

APPENDIX B

PROPERTIES OF 155-MM M30Al PROPELLANT RELEVANT
TO THE RESIDUE PROBLEM

INTRODUCTION

Changes made in both the manufacturing process and the materials used in the M30Al propellant manufacturing process were suggested as causes for the residue problem during the residue investigation. The propellant lots used in the M203 and M203El charges that first gave large amounts of residue were manufactured after August 1977. The process changes that are known to have occurred near this time were:

- 1. A change from batch to continuous (CIN) processing of the nitrocellulose used in the propellant.
- 2. A change in the denaturant used in the ethanol solvent used in propellant manufacture from methanol to benzene/toluene.
- 3. A change in the source of the ethyl centralite used in the propellant.

These or other changes were thought to be responsible for the lower relative quickness (RQ) of the propellant, which was reflected in slightly increased web sizes.

To aid in clarifying the role of the propellant in residue formation, a laboratory program to characterize the M30Al propellant lots was undertaken. Detailed comparisons were made between lot E36 (manufactured before August 1977) and lot 69805 (manufactured after August 1977).

BASIC PROPERTIES

The density and heat of explosion results are given in table B-1. Standard methods were used for the determination of density and heat of explosion.

There is no significant difference in the bulk density. The measured heat of explosion is nearly the same for both propellants but the propellant in lot 69805 has a higher mean — but within one standard deviation from the mean value for the propellant in lot E36. The results were obtained with the propellant at ambient temperature. Differences might be obtained for propellant conditioned at a higher temperature.

Benzene/toluene determinations were made on M30A2 propellant lots 69722 and 69781. These represent lots produced before and after the change in denaturants and were used because a larger web facilitates the extraction.

A methylene chloride extract was taken for use with the gas chromotograph. Only peaks for nitroglycerin and ethyl centralite were observed under conditions for which the measured sensitivity for benzene/toluene was better than 1 ppm.

The denaturants apparently are not retained; however, there is the possibility of an effect on propellant structure during manufacturing. Other measurements described below tend to discount this possibility as well.

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS (ESCA) AND SCANNING ELECTRON MICROSCOPY (SEM)

The ESCA measurements were made on the nitrogen peaks of the nitrogen containing molecules present in the propellant. The results which were consistent with what was expected for 12.6% nitrogen in the nitrocellulose, indicate no discernible difference in the propellant samples.

A typical spectrum is shown in figure B-1. The three peaks at 400, 406, and 408 eV, representing amine nitrogen, nitro-nitrogen, and nitrate ester nitrogen, appear in the ratio of approximately 3:1:2 which is to be expected with nitrocellulose containing 12.6% nitrogen. Spectra of all the samples showed this ratio, indicating that the nitrocellulose has not decomposed to any significant amount in any of the samples. Loss of a few percent of NO_2 could have been easily detected.

SEM pictures were taken of the propellant samples E36 and 69805. Typical SEM photographs of each of the three propellants are shown in figures B-2 through B-4. The microtomed slices used for the ESCA analysis were broken to reveal the internal structure. The SEM photographs revealed that the microtome formed a smeared surface that masked all details of the mixture. The broken surfaces, however, did show the NQ crystals even though instrument resolution was not optimal. Possibly because of the limited number of grains observed (two samples from each of the

three propellants), no obvious difference between the samples could be detected.

The photographs suggest that at least some of the NQ crystals may be hollow, crystalline needles. Perhaps the percentage of such hollow needles is a variable in the manufacturing process. A method of cutting or treating the grains after cutting needs to be developed so that the grains can be photographed end on. This method would facilitate rapid assessment of the NQ crystal morphology and perhaps give better information of the homogeneity of the mix.

X-RAY DIFFRACTION AND NEUTRON SPECTROSCOPY STUDIES OF M30A1 PROPELLANTS

X-ray diffraction, neutron diffraction, and neutron inelastic scattering have been used for the determination of differences in batch and continuous process M30 propellants. Initial x-ray diffraction characterization of samples microtomed from propellant grains showed some differences; however, these were possibly attributable to preferred orientation effects in the samples.

Consequently, neutron techniques which can be employed with as-received grains were used. Neutron diffraction patterns were taken for M30Al grains of lots 69805 and E36. The pattern for the 69805 grain, although exhibiting the same diffraction peaks as for E36 showed a noticeable relative change in the shape of the "background" level, (in neutron diffraction measurements on hydrogeneous materials the background generally is due to real, incoherent scattering from the sample hydrogens). As with recent diffraction studies of a variety of cellulose samples*, this change suggested the possibility of differences in the amorphous component of the polymer in the batch and continous process produced nitrocellulose.

High resolution neutron diffraction and quasi-elastic neutron scattering (QNS) measurements were also performed. The diffraction patterns were analyzed by comparing Bragg peak to background level at the Bragg peak, from least squares fits for six peaks. The QNS

^{*}H. Prask, et al, "Woodpulp Crystal Structure and Its Effect on Nitrocellulose Physical Properties," ARRADCOM Technical Report ARLCD-TR-79031, ARRADCOM, Dover, NJ, September 1980.

measurements provided comparisons of integrated intensitities (over energy) for the between peak regions. Although dramatic differences were not observed, both analyses suggest that hydrogen bonding in the two samples is slightly different, and/or the amorphous component of the NC in the two samples differs.

MECHANICAL PROPERTIES

Certain physical variations can be seen in the grains of multiperforated gun propellants, which are normal for the manufacturing process used, and ocnsist of (1) axial warping, (2) non-symmetric surface distortion relating to the warping, and (3) randomness of performation location, relative to the warping and surface distortion.

Good (lot E36) and bad (lot 69805) Radford lots of M30Al gun propellant were tested in compression at a strain rate of approximately 10 inches/inch/second at an ambient temperature of 11-17°C and at -45°C. The grains were made into test samples having a length-to-diameter ratio of 1; their ends were machined flat and parallel and were as normal to the grain axis as could be expected. The machined ends were coated with graphite prior to testing. A load cell and a LVDT displacement transducer were used to determine stress and strain as functions of time; the outputs were displayed and photographed on a dual beam oscilloscope.

All results are presented in tables B-2 and B-3; typical test records are shown in figures B-5 and B-6. The data of table B-1, since this table is more complete, are shown graphically in figure B-7 and B-8.

Comparisons of the limited data are possible for the good and bad lots of M30Al propellant. It is seen that: (1) differences in the compressive strengths, or in the corresponding strains, for a given set of loading conditions (rate and temperature) are less than the scatter (standard deviations) inherent in the data, and that (2) differences in the compressive Young's moduli at 12°C appear to fall outside the scatter found within the data. This modulus should be less sensitive to the macroscopic physical differences between individual grains described above. It is important to recognize that the M30Al propellant lots have a 3-year difference in packaging dates and that any differences in the moduli could be due to aging.

No significant differences were observed in either compressive strength or corresponding strain between good and bad propellant lots. Any differences noted in Young's modulus between good and bad propellant lots should be regarded as unsubstantiated until further testing can be performed, particularly at higher gain for low strain deformation.

Table B-1. Basic properties of 155 mm M30Al and 8-inch M30A2 propellants

Benzene or toluene concentration $^{\mathtt{c}}$ l ppm $^{\mathtt{l}}$	Heat of explosion $(cal/g)^a$ Density at 20° C $(g/cm^3)^b$	8 in. M30A2 69722 987.9 ± 2.6 98	30A2 69781 980.6 ± 2.0 1.690	155 mm M30A1 E36 968.8 ± 2.0 962 1.673 1	M30A1 69805 962.1 ± 0.5 1.676
	ne or toluene concentration ^c	l ppm	l ppm		

^aDetermined using calorimetry

 $^{
m b}{
m Determined}$ using methods given in MIL-STD-286B

CDetermined using gas chromatography

Table B-2. Compressive mechanical properties of M30Al propellant tested at high rate of strain and $12\,^{\circ}\text{C}$

Lot no.	Young's Modulus (10 ⁶ psi)	Compressive Strength (10 ³ psi)	Strain (Percent)
E36 E36 E36 Mean	2.38 2.28 2.06 2.24 ± 6.0%	13.98 14.20 15.05 14.41 ± 3.2%	1.3 3.5 3.4
69805 69805 69805 69805 Mean	2.04 1.77 2.10 1.82 1.93 ± 6.0%	16.00 14.44 15.46 14.34 15.06 ± 4.6%	2.6 3.3 2.9 3.4

Table B-3. Miscellaneous compressive test data at high rate of strain

Lot no.	Temperature (°C)	Young's Modulus* (10 ⁵ psi)	Compressive strength (10 ³ psi)	Strain (%)
E36	-45	6.56	26.5	0.7
69805	- 45	3.62	25.0	2.2
69805	-45	2.89	29.9	2.7
E36	15	2.73	13.9	1.1
69805	15	2.63	12.6	1.1
	no. E36 69805 69805 E36	no. (°C) E36 -45 69805 -45 69805 -45 E36 15	Lot no. Temperature (°C) Modulus* (10 ⁵ psi) E36 -45 6.56 69805 -45 3.62 69805 -45 2.89 E36 15 2.73	Lot no. Temperature (°C) Modulus* (10 ⁵ psi) strength (10 ³ psi) E36 -45 6.56 26.5 69805 -45 3.62 25.0 69805 -45 2.89 29.9 E36 15 2.73 13.9

^{*}Young's modulus represents the tangent modulus through the origin.

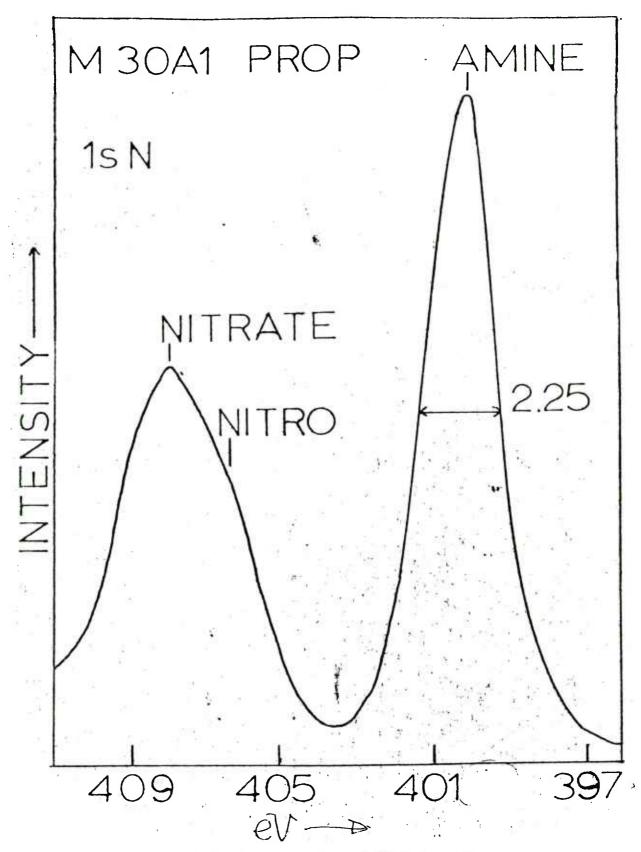


Figure B-1. ESCA spectrum of M30Al propellant

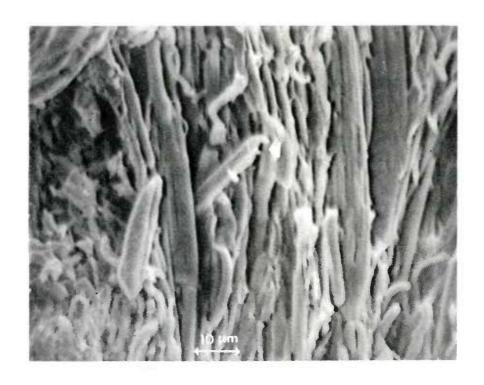


Figure B-2. SEM of M30Al propellant lot E36 at 1000X magnification and 10 $\ensuremath{\mathrm{KV}}$

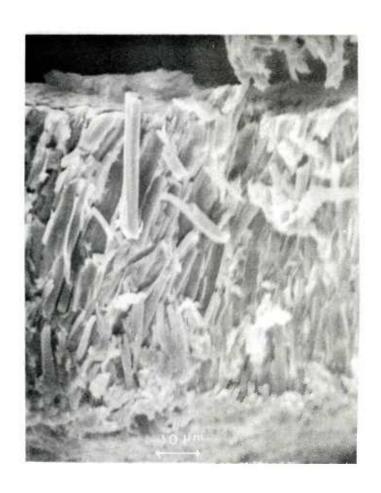
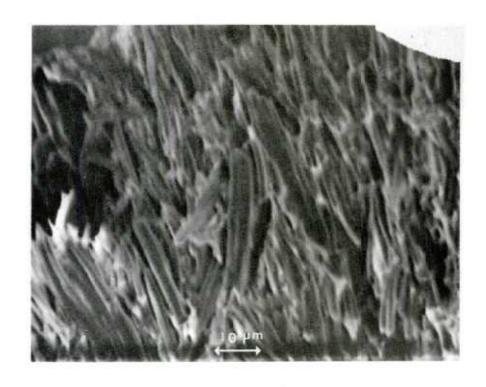


Figure B-3. SEM of M30Al propellant lot E2 at 1000 X magnification and 20 KV



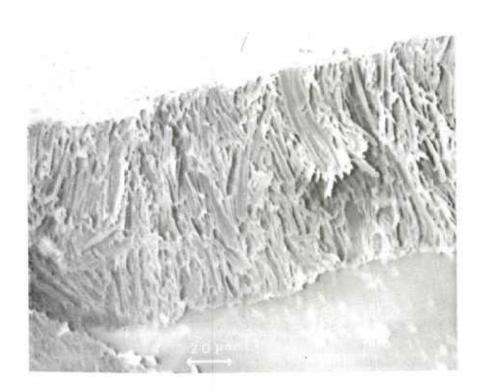
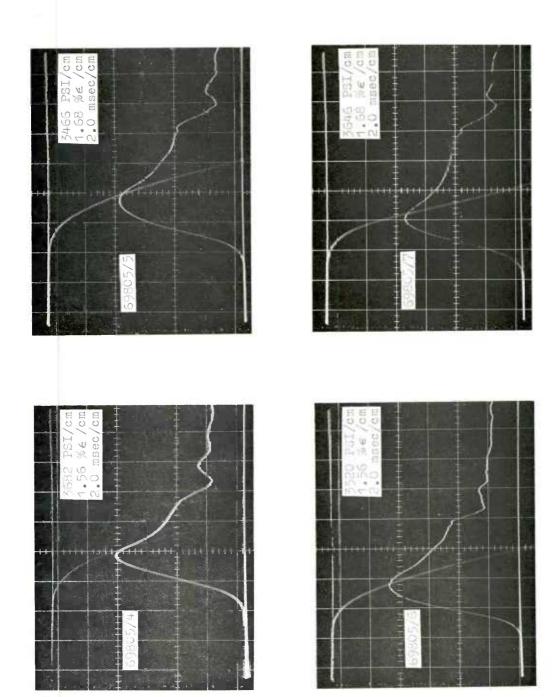
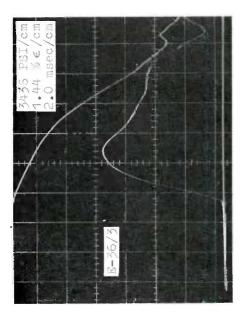
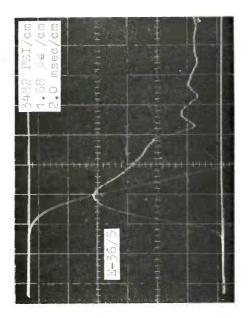


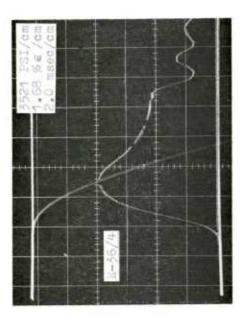
Figure B-4. SEM of M30Al propellant lot 69805 at 1000K and 500K magnification and 10KV



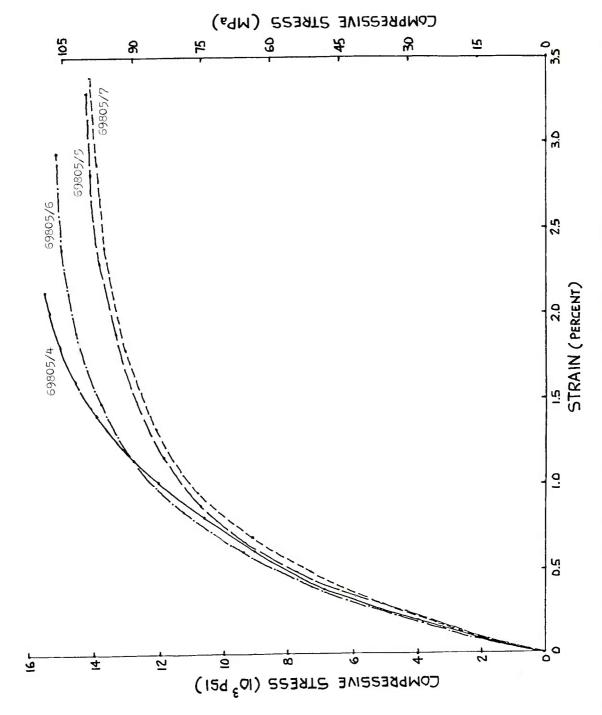
Oscilloscope records of load (lower trace) - displacement (upper trace) for M30Al propellant lot 69805 tested in compression at high rate and $\pm 12\,^{\circ}\mathrm{C}$ Figure B-5.



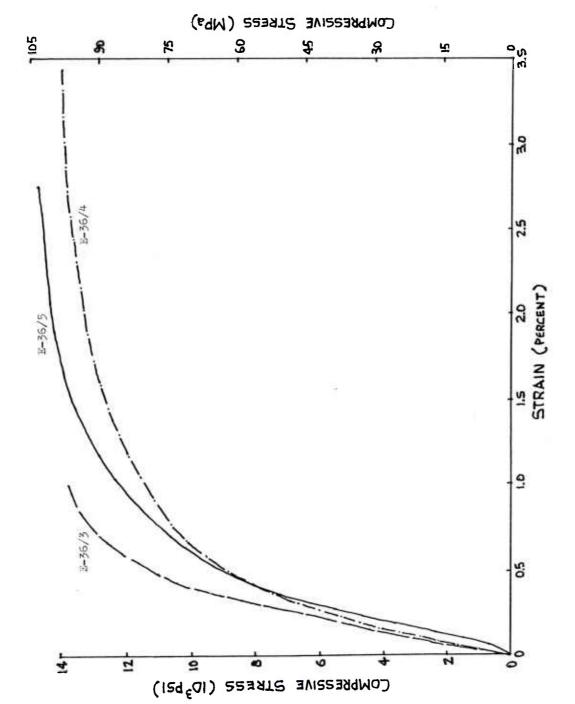




Oscilloscope records of load (lower trace) - displacement (upper trace) for M30Al propellant lot E36 tested in compression at high rate and +12°C Figure B-6.



High rate stress-strain curves for M30Al propellant lot 69805 truncated at the maximum stress; strain rate - 10 inches/inch/second; temperature - $12\,^{\circ}\mathrm{C}$ Figure B-7.



High rate stress-strain curves for M30Al propellant lot E36 truncated at the maximum stress; strain rate - 10 inches/inch/second; temperature - $12\,^{\circ}\mathrm{C}$ Figure B-8.

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Crane, IN 47522